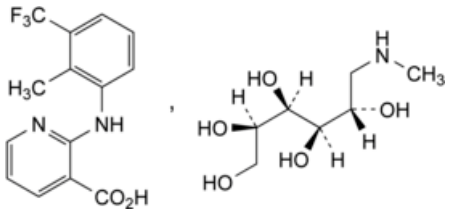
Edition: BP 2025 (Ph. Eur. 11.6 update)

Flunixin Meglumine

General Notices

(Flunixin Meglumine for Veterinary Use, Ph. Eur. monograph 1696)



$C_{21}H_{28}F_3N_3O_7$ 491.5 *42461-84-7*

Action and use

Cyclo-oxygenase inhibitor; analgesic; anti-inflammatory.

Ph Eur

DEFINITION

2-[[2-Methyl-3-(trifluoromethyl)phenyl]amino]pyridine-3-carboxylic acid, 1-deoxy-1-(methylamino)-D-glucitol.

Content

99.0 per cent to 101.0 per cent (dried substance).

CHARACTERS

Appearance

White or almost white, crystalline powder.

Solubility

Freely soluble in water and in methanol, practically insoluble in acetone.

IDENTIFICATION

A. Specific optical rotation (2.2.7): -12.0 to -9.0 (dried substance), determined on solution S (see Tests).
B. Infrared absorption spectrophotometry (2.2.24).

Comparison *flunixin meglumine CRS*.

TESTS

Solution S

Dissolve 2.50 g in *carbon dioxide-free water R* and dilute to 50.0 mL with the same solvent.

Appearance of solution

Solution S is clear (*2.2.1*) and not more intensely coloured than reference solution Y_7 (*2.2.2, Method II*).

pH (*2.2.3*)

7.0 to 9.0 for solution S.

Related substances

Liquid chromatography (*2.2.29*).

Test solution Dissolve 50.0 mg of the substance to be examined in the mobile phase and dilute to 10.0 mL with the mobile phase.

Reference solution (a) Dissolve 5.0 mg of *flunixin impurity B CRS* in 1.0 mL of the test solution and dilute to 50.0 mL with the mobile phase.

Reference solution (b) Dissolve 5.0 mg of *2-chloronicotinic acid R* (impurity A) in the mobile phase and dilute to 50.0 mL with the mobile phase. To 2.0 mL of this solution add 2.0 mL of reference solution (a) and dilute to 20.0 mL with the mobile phase.

Reference solution (c) Dissolve 50 mg of *flunixin impurity C CRS* in the mobile phase and dilute to 100 mL with the mobile phase.

Column:

— *size*: *l* = 0.125 m, Ø = 4.0 mm,

— *stationary phase*: *octadecylsilyl silica gel for chromatography R* (5 µm).

Mobile phase Mix 300 volumes of *water R* and 700 volumes of *acetonitrile R*, and add 0.25 volumes of *phosphoric acid R*.

Flow rate 1.0 mL/min.

Detection Spectrophotometer at 254 nm.

Injection 10 µL.

Run time 5 times the retention time of flunixin.

Relative retention With reference to flunixin (retention time = about 3.1 min): impurity A = about 0.4; impurity C = about 0.6; impurity B = about 0.7; impurity D = about 4.2.

System suitability Reference solution (a):

— *resolution*: minimum 3.5 between the peaks due to impurity B and flunixin.

Limits:

— *correction factor*: for the calculation of content, multiply the peak area of impurity C by 1.9,

— *impurity A*: not more than the area of the corresponding peak in the chromatogram obtained with reference solution (b) (0.2 per cent),

— *impurity B*: not more than the area of the corresponding peak in the chromatogram obtained with reference solution (b) (0.2 per cent),

— *impurities C, D*: for each impurity, not more than the area of the peak due to flunixin in the chromatogram obtained with reference solution (b) (0.2 per cent),

— *any other impurity*: for each impurity, not more than the area of the peak due to flunixin in the chromatogram obtained with reference solution (b) (0.2 per cent),

— *total*: not more than 2.5 times the area of the peak due to flunixin in the chromatogram obtained with reference solution (b) (0.5 per cent),

— *disregard limit*: 0.25 times the area of the peak due to flunixin in the chromatogram obtained with reference solution (b) (0.05 per cent).

Loss on drying (*2.2.32*)

Maximum 0.5 per cent, determined on 1.000 g by drying in an oven at 105 °C for 4 h.

Sulfated ash (*2.4.14*)

Maximum 0.1 per cent, determined on 1.0 g.

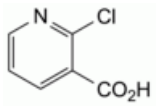
ASSAY

Dissolve 0.175 g in 50 mL of *anhydrous acetic acid R*. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*).

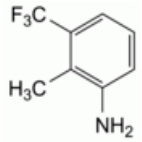
1 mL of *0.1 M perchloric acid* is equivalent to 24.57 mg of $C_{21}H_{28}F_3N_3O_7$.

IMPURITIES

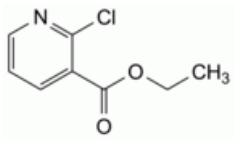
Specified impurities *A, B, C, D.*



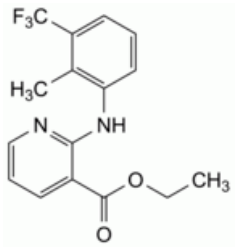
A. 2-chloropyridine-3-carboxylic acid,



B. 2-methyl-3-(trifluoromethyl)aniline,



C. ethyl 2-chloropyridine-3-carboxylate,



D. ethyl 2-[[2-methyl-3-(trifluoromethyl)phenyl]amino]pyridine-3-carboxylate.
